Slag Samples - From April 12, 2011 Event at WTI

HRG / Ralph Roper, PhD, PE / April 29, 2011

Sample Collection:

The samples were collected two days after the event after the kiln had cooled down. They were generally from a "donut" like accumulation of slag that was about 5 -feet down from the feed end, and was about 4 feet wide. It was up to about 2 feet thick, but the thickness was not uniform. Kevin collected the samples after jackhammering them loose.

General Appearance of Slag Samples:

The slag exhibited at least two very distinct layers - a thin orangy top layer and a thick black bottom layer. As shown in Figure 1, some of the samples had a third layer of gray slag sandwiched between the top orangish layer the bottom blackish layer.

Figure 1 - Photograph of a sample from the do-nut slag showing three layers

As shown in Figure 2, one of the slag samples was thin and flat and probably came from further down the barrel of the kiln beyond the donut accumulation. The underneath side appeared to have accumulations of metallic aluminum (see Figure 3)

Figure 2 - Photograph of a Slag Sample further down the barrel of the kiln

Figure 3 - Photograph of a bottom side of slag shown in Figure 2

Slag Composite Samples

Various chunks of slag were used to make composite samples representative of the three layers previously shown in Figure 1. In addition, a fourth composite was made of the metallic-looking material shown in Figure 3. The materials used for the four composites are shown in Figure 4.

Fig 4-1 Composite Sample 1

Fig 4-12 Composite Sample 2

Fig 4-3 Composite Sample 3

Fig 4-4 Composite Sample 4

Results from XRF Analyses

The XRF results are summarized in Table 1. The results are only approximate in that the instrument measures the element concentrations, then converts them mathematically to oxides, then scales the results to equal 100%. In actuality, the constituents were not all oxides. The presence of Lanthanum was a surprise to the analysts. The slag composites were mainly silicates and aluminates with lesser amounts of iron, lanthanum and calcium.

Table 1 - Results from SRF Analyses (%)

	Comp 1	Comp 2	Comp 3	Comp 4	MW
Acidic Oxides					
SiO2	43.88	45.92	47.59	43.98	60
Al2O3	36.45	36.49	29.64	26.13	102
P2O5	1.23	1.11	1.48	1.69	142
La2O3	3.09	2.97	2.19	2.1	326
TiO2	1.71	3.25	2.05	2.32	80
Basic Oxides					
Fe2O3	4.05	3.30	6.92	5.8	104
CaO	2.67	2.19	3.71	2.95	56
MgO	1.51	1.34	1.94	2.26	40
CuO	0.50	0.10	0.12	0	80
Na2O	1.24	1.34	1.40	5.67	62
K2O	0.42	0.42	1.01	5.41	94
BaO	1.07	0.11	0.43	0.07	153
NiO	0.47	0.45	0.35	0.39	75

Calculation of Slag Basicity

Basicity = (A - 3xB)/2xC where:

A = No. moles of basic oxides
B = No. moles of Al2O3 + P2O5
C = No. moles of SiO2 + TiO2

A basicity of 1 represents a neutral slag. Slag is more "basic" as the basicity increases above unite, and becomes more acidic as it decreases below unity. As calculated below, the slag is very acidic.

A =	0.168349	0.138388	0.223591	0.319610
B =	0.366015	0.365562	0.301011	0.268078
C =	0.752708	0.805958	0.818792	0.762000
Basicity =	**-0.34989**	**-0.38617**	**-0.27816**	**-0.18464**

Thermographic Analysis

Thermographic analyses were done on each of the four samples. The results are attached. The initial phase of each test (blue part of the curve) were done under nitrogen gas, and then the atmosphere was oxygen during the second phase (red part of the curve). With the exception of composite sample 4, all of the thermographic analyses showed an initial reduction in weight followed by an overall net increase in weight when oxygen was added with increasing temperatures. Composite sample 4 was from a thin flat slag sample that probably came from further down the barrel of the kiln. As can be seen in Figure 5, all four of the samples were orangish in color after the TGA analysis.

One interpretation of the TGA analysis is the the net weight gain could be from the oxidation of ferrous iron, FeO to ferric iron, Fe2O3. This was further supported by the orangish change in slag color. In fact, the blackish color of the slag in general suggested the slag was being formed under reducing conditions rather than under oxidatizing conditions.

Figure 5 Color Slag samples before and after TGA TGA analysis

XRD Analyses

The results from the XRD analyses are attached and summarized in Table 2. As expected, the compounds were mainly aluminosilicates or silicates.

Table 2 - Results from XRD Analyses (%)

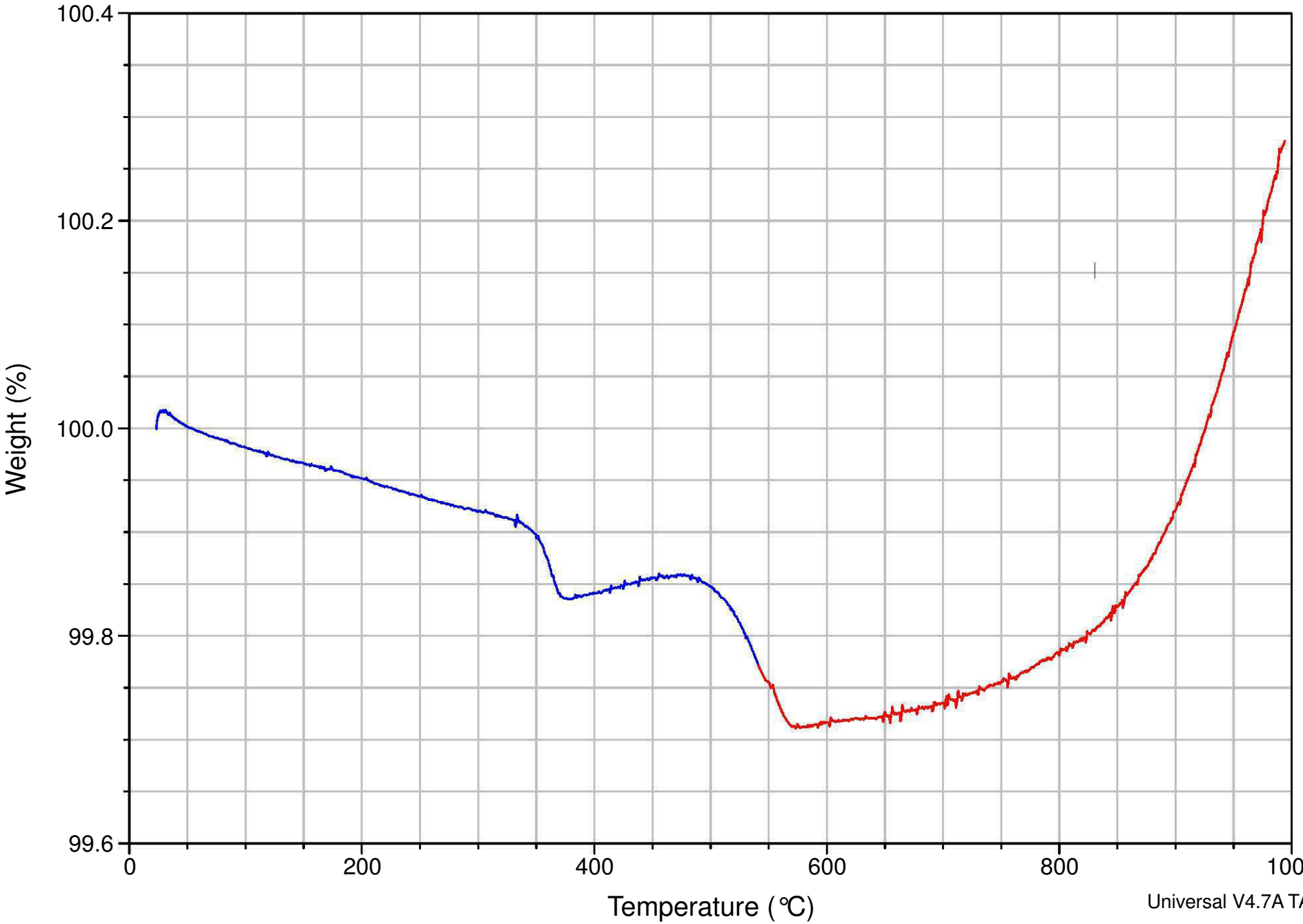
		Comp 1	Comp 2	Comp 3	Comp 4
Mullite	$Al_{2.5}Si_{0.85}O_{4.93}$	34.6	45.2	23.3	7.0
Anorthite	$CaAl_2Si_2O_8$	5.1	6.2	21.1	-
Quartz	SiO_2	2.8	2.3	5.3	3.8
Cristobalite	SiO_2	2.2	5.5	2.8	-
Corundun	Al_2O_3	1.5	2.1	3.2	-
K-Mg-Al-Si Oxide	$KMg_2Al_5Si_4O_{18}$	0.8	6.3	6.9	-
Spinel	$MgAl_2O_4$	-	-	7.2	11.2
Albite	$NaAlSi_3O_8$	-	-	-	12.0
Anorthoclase	$K_{0.334}Na_{0.686}Ca_{0.006}Al_{0.987}Si_{2.994}O_8$	-	-	-	11.5
Nepheline	$K_{0.8}Na_3Al_{3.6}Si_{0.2}(SiO_4)_4$	-	-	-	11.0
Amorphous	-	53.0	32.4	30.1	43.5

Sample: 6252 WTI comp 1
Size: 35.1320 mg
Method: Ramp
Comment: 20 degrees per minute

TGA

File: \\...\6252 WTI comp 1.001

Run Date: 27-Apr-2011 13:39
Instrument: TGA Q500 V20.10 Build 36

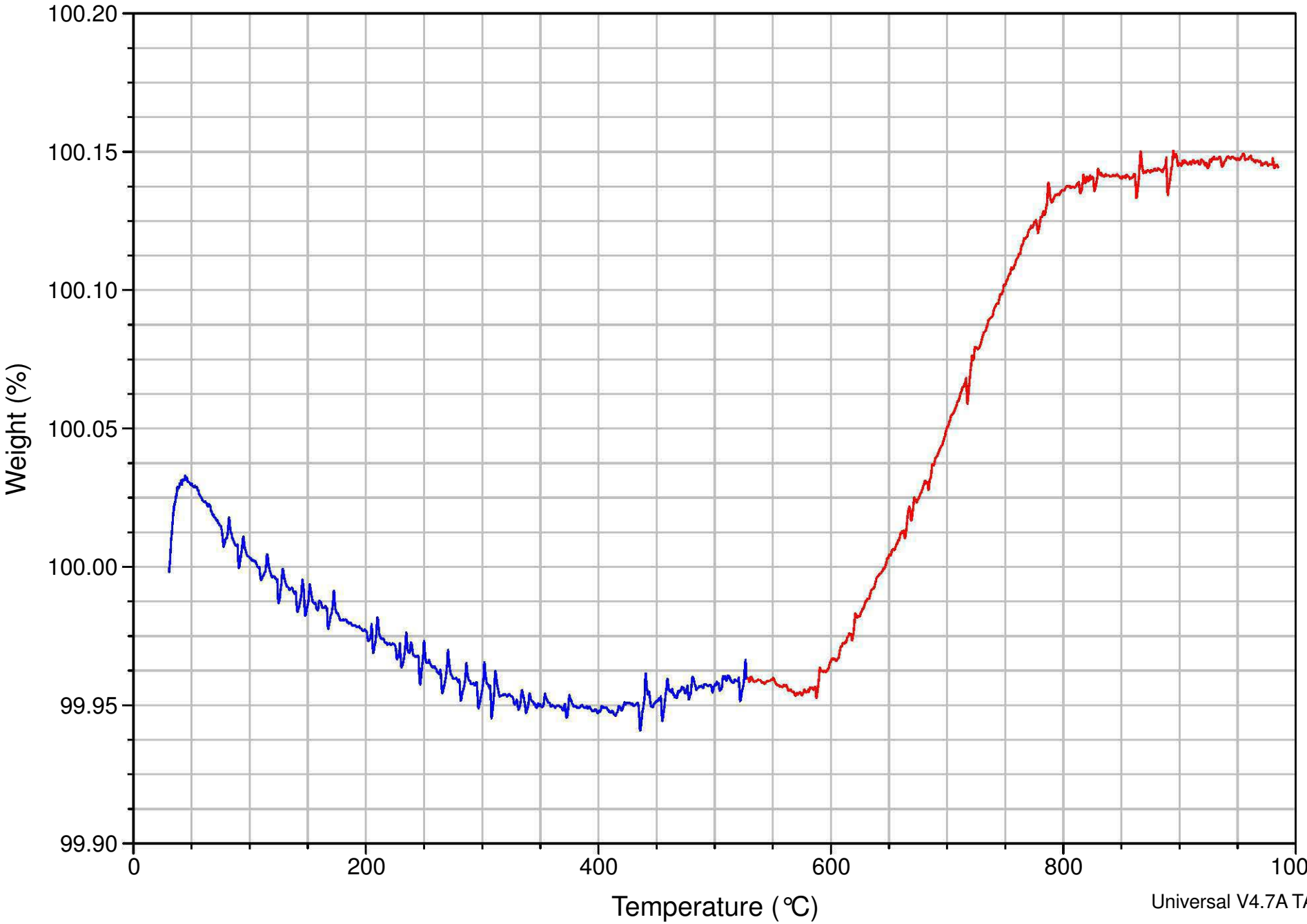


Sample: 6252 WTI comp 2
Size: 24.6910 mg
Method: Ramp
Comment: 20 degrees per minute

TGA

File: \\...\6252 WTI comp 2.001

Run Date: 27-Apr-2011 14:40
Instrument: TGA Q500 V20.10 Build 36

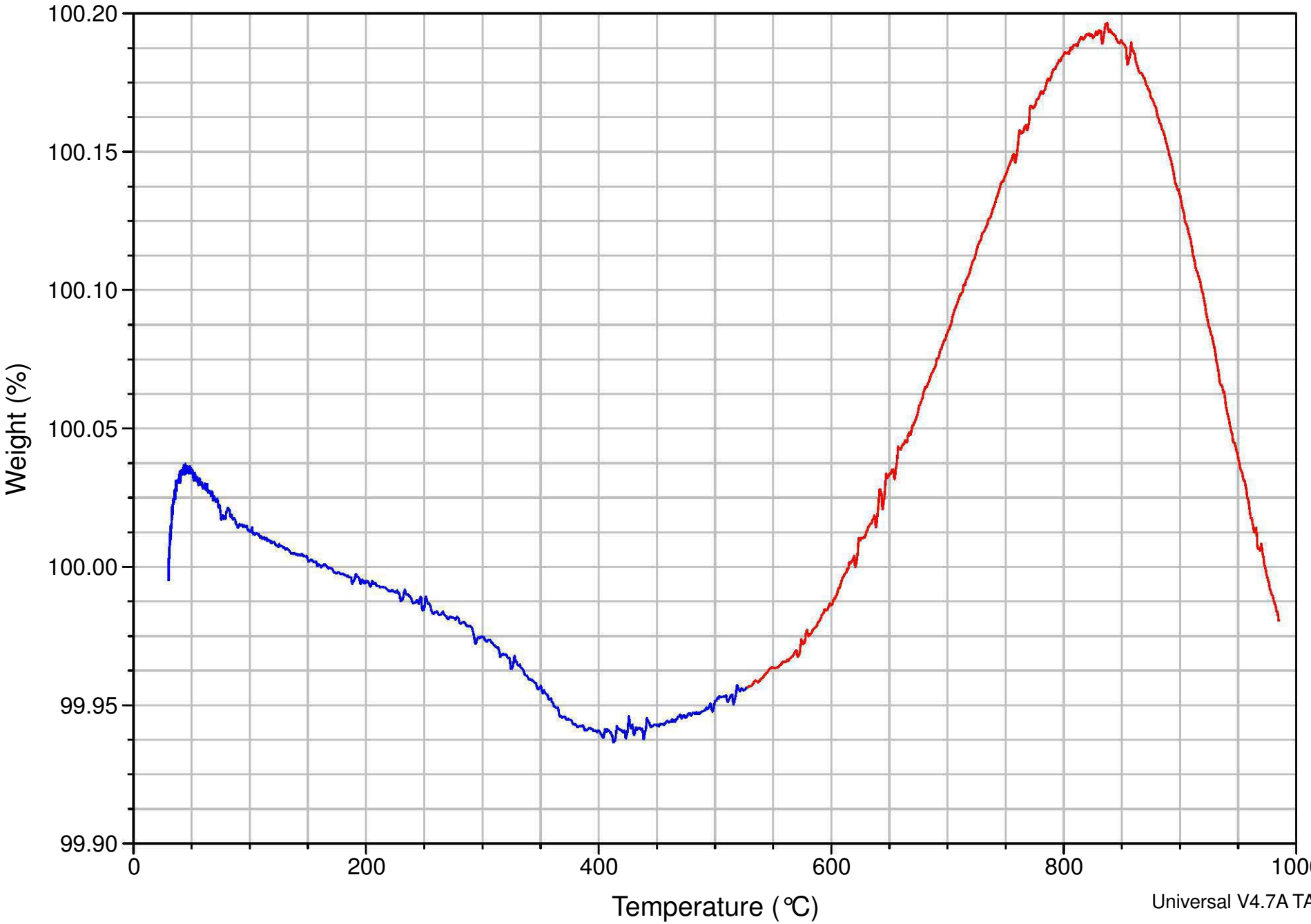


Sample: 6252 WTI comp 3
Size: 27.3620 mg
Method: Ramp
Comment: 20 degrees per minute

TGA

File: \\...\6252 WTI comp 3.001

Run Date: 27-Apr-2011 15:11
Instrument: TGA Q500 V20.10 Build 36

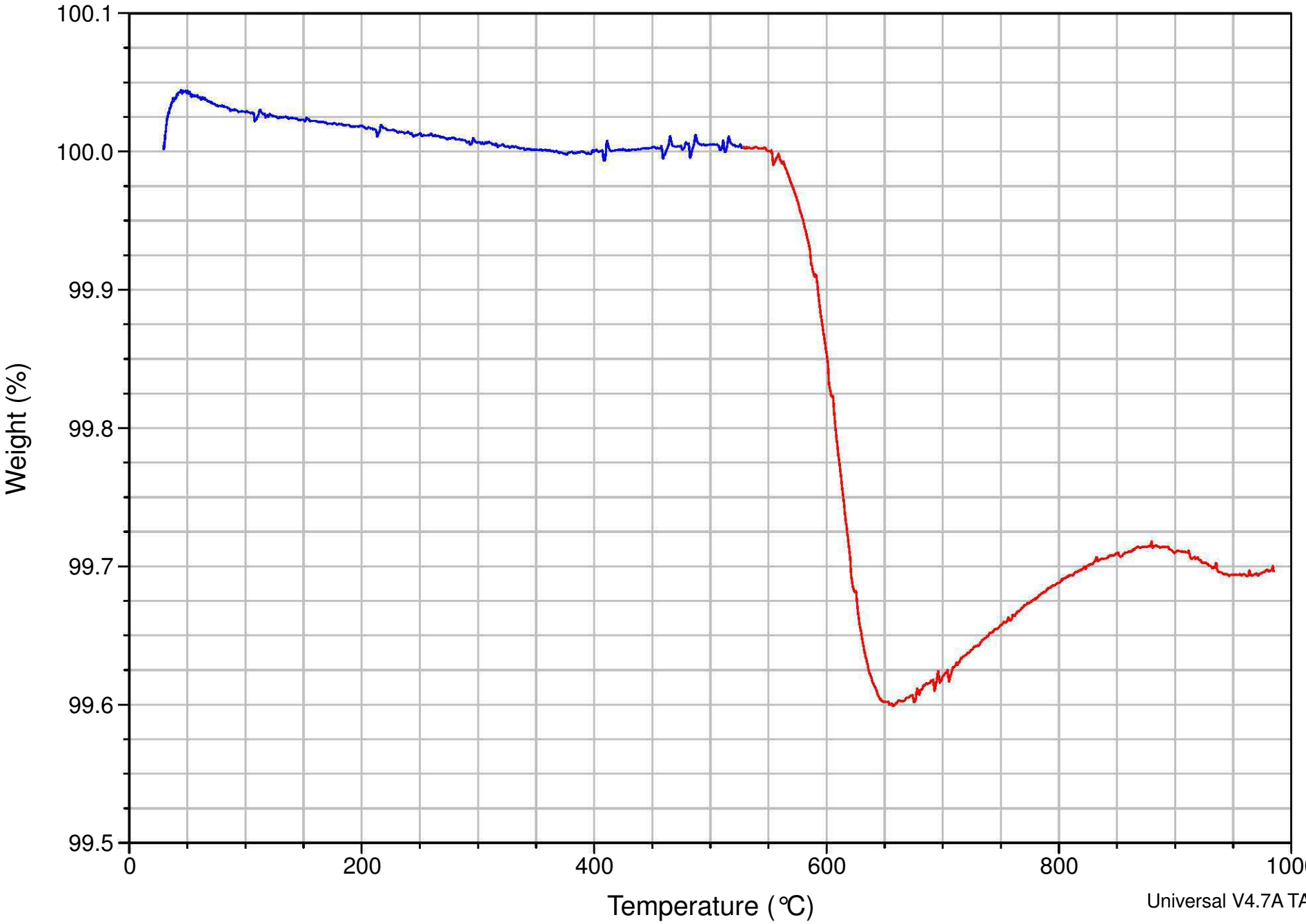


Sample: 6252 WTI comp 4
Size: 22.3780 mg
Method: Ramp
Comment: 20 degrees per minute

TGA

File: \\...\6252 WTI comp 4.001

Run Date: 27-Apr-2011 15:43
Instrument: TGA Q500 V20.10 Build 36



6252 comp 1

FILE: [6252_comp_1.raw] 6252 comp 1 - Quantitative Scan
SCAN: 5.0/70.0/0.02/2(sec), Cu(30kV,15mA), I(p)=2697, 04/26/11 02:14p
PROC: [WPF Control File]

☑ K-alpha2 Peak Present
☑ Apply Anomalous Scattering
☑ Use Isotropic B Value Only

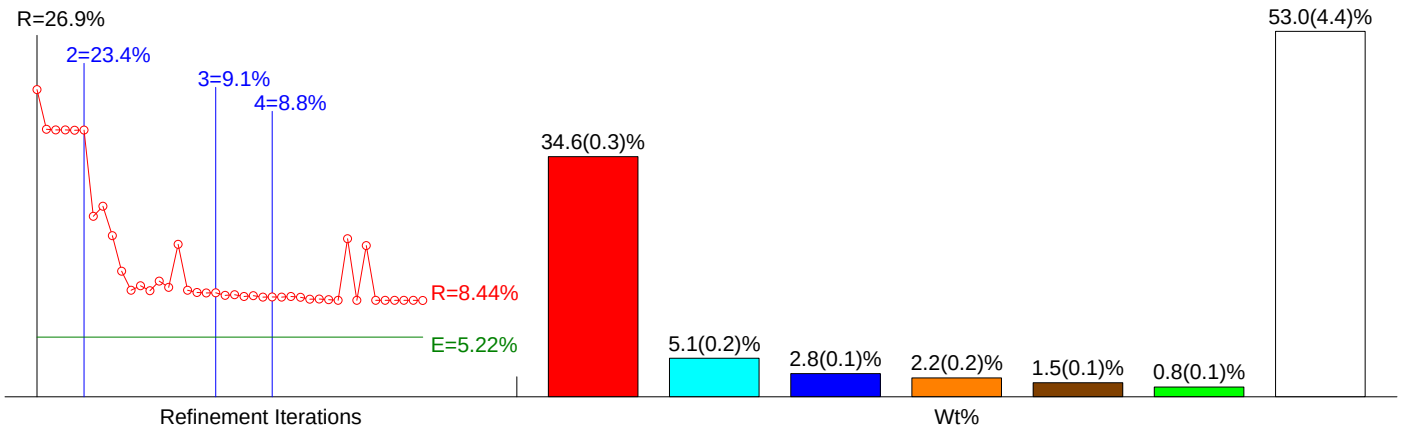
[Diffractometer LP] Two-Theta Range of Fit = 5.0 - 70.0(deg)
☑ Specimen Displacement - Cos(Theta) = -0.094158(0.003877)
☐ Monochromator Correction for LP Factor = 1.0
☐ K-alpha2/K-alpha1 Intensity Ratio = 0.5

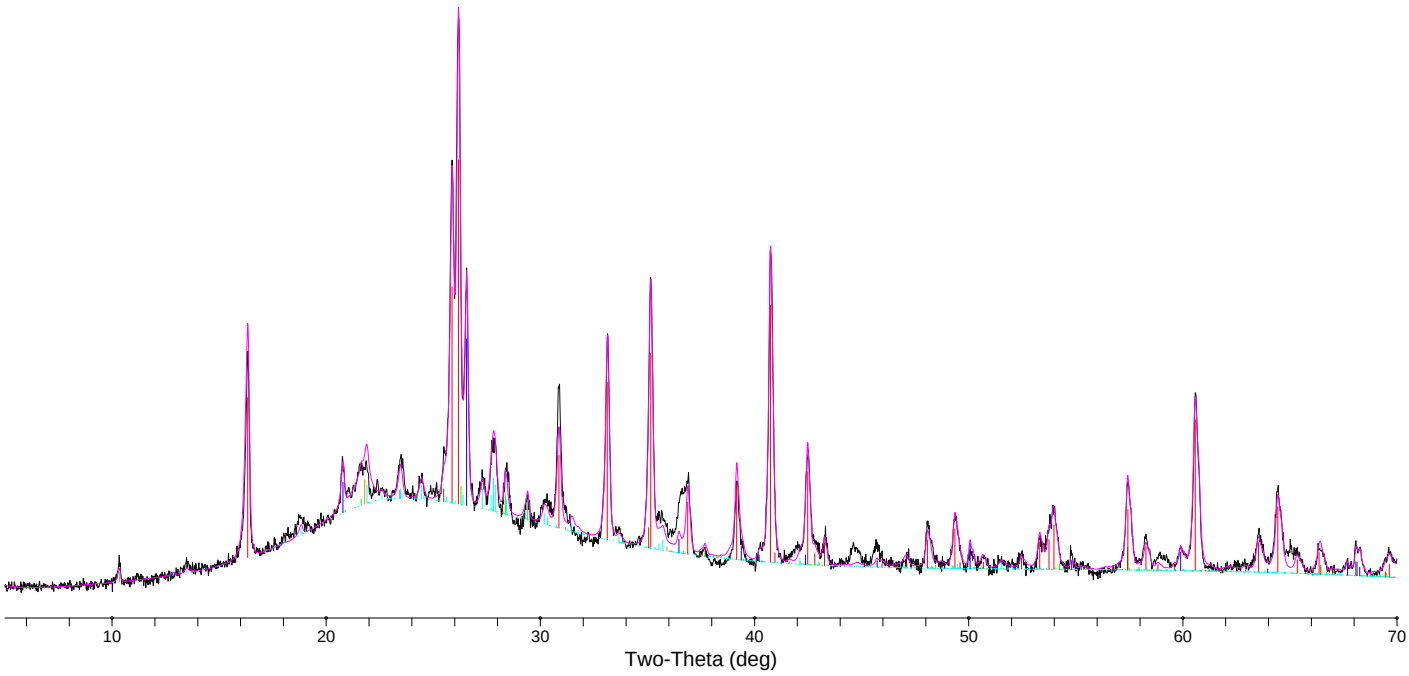
Profile Shape Function (PSF) for All Phases: Pearson-VII, Polynomial(3), Lambda=1.54059Å (Cu/K-alpha1)

Phase ID (6)	Source	I/Ic	Wt%	#L
Mullite - $Al_{2.15}Si_{0.85}O_{4.93}$	PDF#04-008-9529	0.84(0%)	34.6 (0.3)	48
Anorthite - $CaAl_2Si_2O_8$	PDF#04-011-2883	0.61(0%)	5.1 (0.2)	864
Quartz - SiO_2	PDF#04-002-3600	4.29(0%)	2.8 (0.1)	54
Cristobalite - SiO_2	PDF#04-008-7638	4.98(0%)	2.2 (0.2)	66
Corundun - Al_2O_3	PDF#04-007-5143	1.05(0%)	1.5 (0.1)	13
Potassium Magnesium Aluminum Silicon Oxide - $KMg_2Al_5Si_4O_{18}$	PDF#04-009-2350	1.21(0%)	0.8 (0.1)	67
Others + Amorphous			53.0 (4.4)	

XRF(Wt%): Ca=0.7%, K=0.1%, Si=32.5%, Al=15.4%, Mg=0.1%, O=51.2%

NOTE: Fitting Halted at Iteration 42(4): R=8.44% (E=5.22%, R/E=1.62, P=49, EPS=0.5)





6252 comp 2

FILE: [6252_comp_2.raw] 6252 comp 2 - Quantitative Scan
SCAN: 5.0/70.0/0.02/2(sec), Cu(30kV,15mA), I(p)=3072, 04/26/11 06:05p
PROC: [WPF Control File]

☑ K-alpha2 Peak Present
☑ Apply Anomalous Scattering
☑ Use Isotropic B Value Only

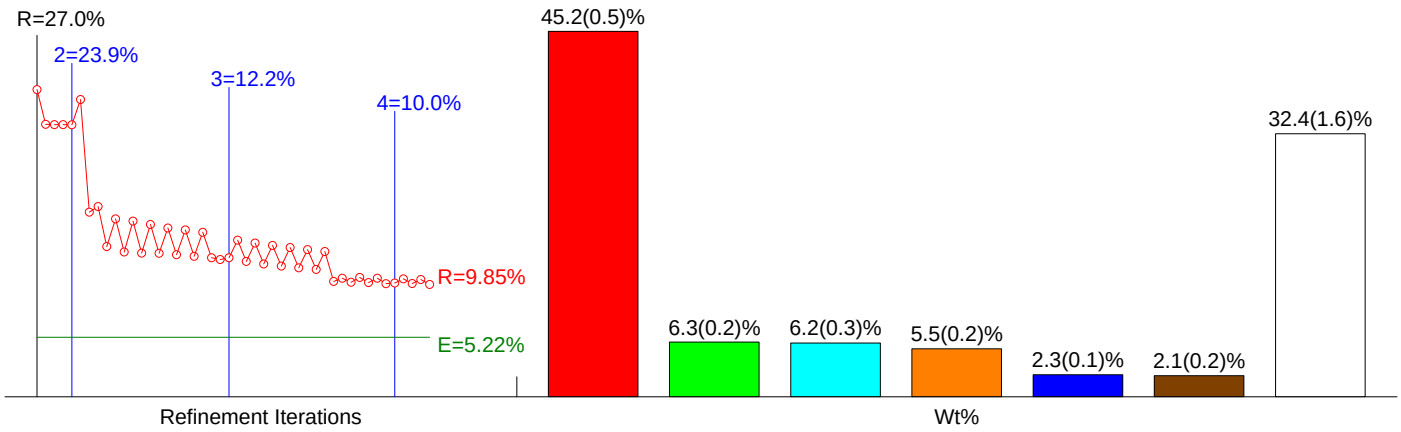
[Diffractometer LP] Two-Theta Range of Fit = 5.0 - 70.0(deg)
☑ Specimen Displacement - Cos(Theta) = -0.103193(0.003822)
☐ Monochromator Correction for LP Factor = 1.0
☐ K-alpha2/K-alpha1 Intensity Ratio = 0.5

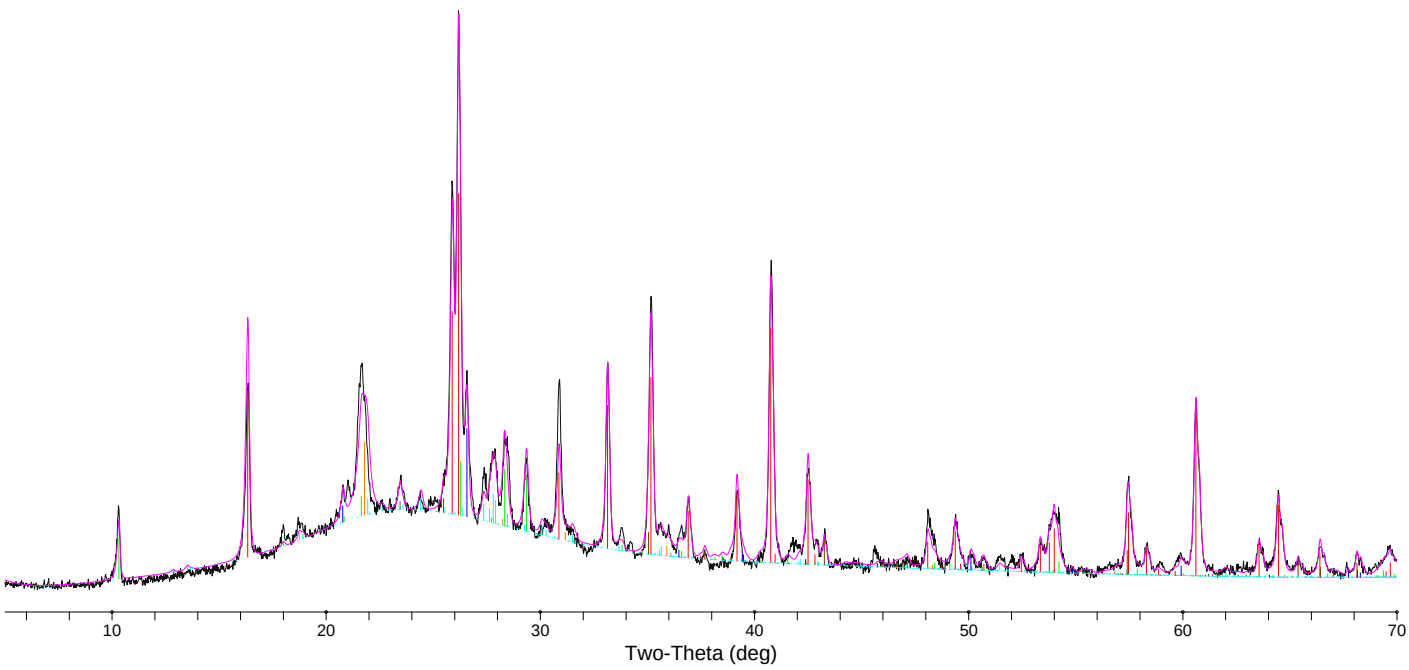
Profile Shape Function (PSF) for All Phases: Pearson-VII, Polynomial(3), Lambda=1.54059Å (Cu/K-alpha1)

Phase ID (6)	Source	I/Ic	Wt%	#L
Mullite - $Al_{2.15}Si_{0.85}O_{4.93}$	PDF#04-008-9529	0.84(0%)	45.2 (0.5)	48
Potassium Magnesium Aluminum Silicon Oxide - $KMg_2Al_5Si_4O_{18}$	PDF#04-009-2350	1.21(0%)	6.3 (0.2)	68
Anorthite - $CaAl_2Si_2O_8$	PDF#04-011-2883	0.61(0%)	6.2 (0.3)	861
Cristobalite - SiO_2	PDF#04-008-7638	4.98(0%)	5.5 (0.2)	66
Quartz - SiO_2	PDF#04-002-3600	4.30(0%)	2.3 (0.1)	54
Corundun - Al_2O_3	PDF#04-007-5143	1.05(0%)	2.1 (0.2)	13
Others + Amorphous			32.4 (1.6)	

XRF(Wt%): Ca=0.9%, K=0.4%, Si=26.7%, Al=21.3%, Mg=0.5%, O=50.2%

NOTE: Fitting Halted at Iteration 46(4): R=9.85% (E=5.22%, R/E=1.89, P=51, EPS=0.5)





6252 comp 3

FILE: [6252_comp_3.raw] 6252 comp 3 - Quantitative Scan
SCAN: 5.0/70.0/0.02/2(sec), Cu(30kV,15mA), I(p)=1795, 04/26/11 07:57p
PROC: [WPF Control File]

☑ K-alpha2 Peak Present
☑ Apply Anomalous Scattering
☑ Use Isotropic B Value Only

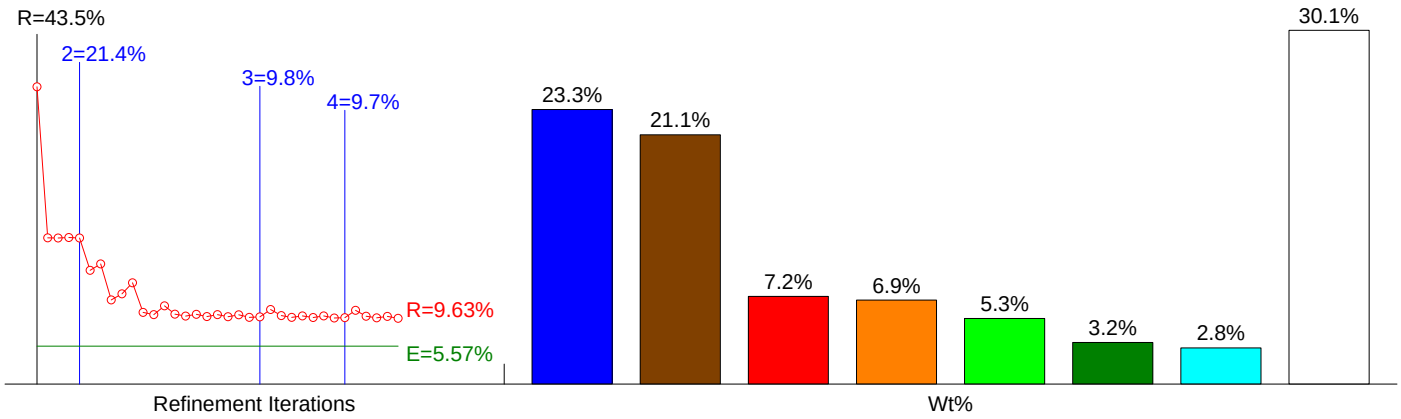
[Diffractometer LP] Two-Theta Range of Fit = 5.0 - 70.0(deg)
☑ Specimen Displacement - Cos(Theta) = -0.141513(0.004412)
☐ Monochromator Correction for LP Factor = 1.0
☐ K-alpha2/K-alpha1 Intensity Ratio = 0.5

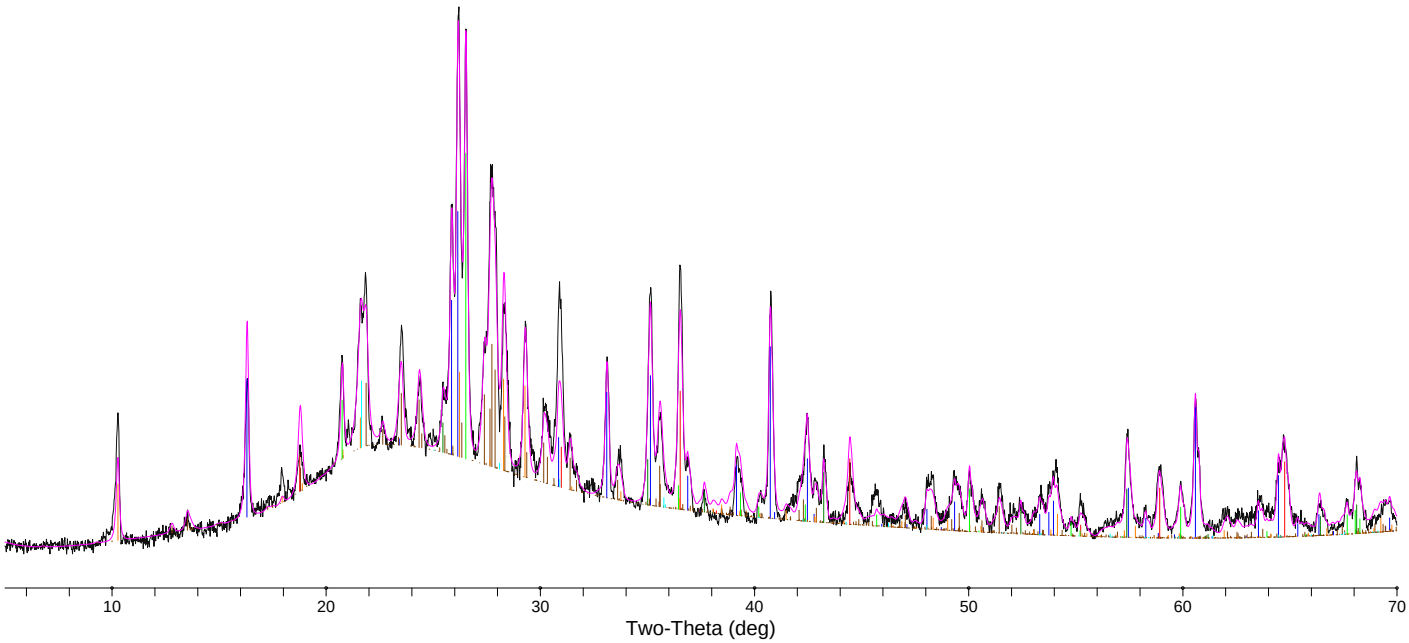
Profile Shape Function (PSF) for All Phases: Pearson-VII, Polynomial(3), Lambda=1.54059Å (Cu/K-alpha1)

Phase ID (7)	Source	I/Ic	Wt%	#L
Mullite - $Al_{2.15}Si_{0.85}O_{4.93}$	PDF#04-008-9529	0.85(0%)	23.3 (0.4)	48
Anorthite - $CaAl_2Si_2O_8$	PDF#04-011-2883	0.61(0%)	21.1 (0.4)	861
Spinel - $MgAl_2O_4$	PDF#04-008-0807	1.68(0%)	7.2 (0.2)	12
Potassium Magnesium Aluminum Silicon Oxide - $KMg_2Al_5Si_4O_{18}$	PDF#04-009-2350	1.22(0%)	6.9 (0.2)	68
Quartz - SiO_2	PDF#04-002-3600	4.31(0%)	5.3 (0.1)	54
Corundun - Al_2O_3	PDF#04-007-5143	1.05(0%)	3.2 (0.2)	13
Cristobalite - SiO_2	PDF#04-008-7638	4.99(0%)	2.8 (0.2)	66
Others + Amorphous			30.1 (1.4)	

XRF(Wt%): Ca=3.0%, K=0.4%, Si=26.2%, Al=19.1%, Mg=1.8%, O=49.4%

NOTE: Fitting Halted at Iteration 35(4): R=9.63% (E=5.57%, R/E=1.73, P=56, EPS=0.5)





6252 comp 4

FILE: [6252_comp_4.raw] 6252 comp 4 - Quantitative Scan
SCAN: 5.0/70.0/0.02/2(sec), Cu(30kV,15mA), I(p)=1535, 04/26/11 09:48p
PROC: [WPF Control File]

☑ K-alpha2 Peak Present
☑ Apply Anomalous Scattering
☑ Use Isotropic B Value Only

[Diffractometer LP] Two-Theta Range of Fit = 5.0 - 70.0(deg)
☑ Specimen Displacement - Cos(Theta) = -0.081836(0.007971)
☐ Monochromator Correction for LP Factor = 1.0
☐ K-alpha2/K-alpha1 Intensity Ratio = 0.5

Profile Shape Function (PSF) for All Phases: Pearson-VII, Polynomial(3), Lambda=1.54059Å (Cu/K-alpha1)

Phase ID (6)	Source	I/Ic	Wt%	#L
Albite - $NaAlSi_3O_8$	PDF#04-007-5092	0.65(0%)	12.0 (0.8)	282
Anorthoclase - $K_{0.334}Na_{0.686}Ca_{0.008}Al_{0.987}Si_{2.994}O_8$	PDF#04-013-2164	0.55(0%)	11.5 (1.2)	165
Spinel - $MgAl_2O_4$	PDF#04-008-0798	1.79(0%)	11.2 (0.3)	12
Nepheline - $K_{0.8}Na_3Al_{3.8}Si_{0.2}(SiO_4)_4$	PDF#04-013-5604	1.00(0%)	11.0 (0.4)	240
Mullite - $Al_{2.16}Si_{0.84}O_{4.92}$	PDF#04-008-9531	0.84(0%)	7.0 (0.4)	48
Silicon Oxide - SiO_2	PDF#04-001-9367	4.29(0%)	3.8 (0.1)	54
Others + Amorphous			43.5 (4.2)	

XRF(Wt%): Ca=0.0%, K=1.1%, Si=32.7%, Al=11.4%, Mg=1.9%, Na=3.0%, O=49.9%

NOTE: Fitting Halted at Iteration 33(4): R=11.69% (E=5.45%, R/E=2.15, P=56, EPS=0.5)